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#### (54) Title: SALINE SOLUBLE INORGAL

#### (57) Abstract

A saline soluble fiber is disclosed that is highly refractory. A vacuum cast preform of the fibre has a shrinkage of 3.5 % or less when exposed to 1260 °C for 24 hours. The fibre may comprise CaO, SiO<sub>2</sub>, MgO, optionally ZrO<sub>2</sub>, optionally less than 0.75 mol % Al<sub>2</sub>O<sub>3</sub>, any incidental impurities amounting to less than 2 mol % in total, and in which the SiO<sub>2</sub> excess (defined as the amount of SiO<sub>2</sub> calculated as remaining after the above named constituents are crystallised as silicates) exceeds 21.8 mol %, with the proviso that, if the amount of CaO is greater than the sum of the amount of MgO and twice the amount of ZrO2 the calculated ratio of diopside to wollastonite does not lie in the range 1.8 to 5.25. Such fibres are usable at elevated temperatures where refractoriness is of importance and their solubility in saline solution may make the fibres safer than non-soluble fibres.

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## SALINE SOLUBLE INORGANIC FIBRES

This invention relates to saline soluble, non-metallic, amorphous, inorganic oxide, refractory fibrous materials. The invention particularly relates to glassy fibres having silica as their principal constituent.

Inorganic fibrous materials are well known and widely used for many purposes (e.g. as thermal or acoustic insulation in bulk, mat, or blanket form, as vacuum formed shapes, as vacuum formed boards and papers, and as ropes, varns or textiles; as a reinforcing fibre for building materials; as a constituent of brake blocks for vehicles). In most of these applications the properties for which inorganic fibrous materials are used require resistance to heat, and often resistance to aggressive chemical environments.

Inorganic fibrous materials can be either glassy or crystalline. Asbestos is an inorganic fibrous material one form of which has been strongly implicated in respiratory disease.

It is still not clear what the causative mechanism is that relates some asbestos with disease but some researchers believe that the mechanism is mechanical and size related. Asbestos of a critical size can pierce cells in the body and so, through long and repeated cell injury, have a bad effect on health. Whether this mechanism is true or not regulatory agencies have indicated a desire to categorise any inorganic fibre product that has a respiratory fraction as hazardous, regardless of whether there is any evidence to support such categorisation. Unfortunately for many of the applications for which inorganic fibres are used, there are no realistic substitutes.

Accordingly there is a demand for inorganic fibres that will pose as little risk as possible (if any) and for which there are objective grounds to believe them safe.

A line of study has proposed that if inorganic fibres were made that were sufficiently soluble in physiological fluids that their residence time in the human body was short; then damage would not occur or at least be minimised. As the risk of asbestos linked disease appears to depend very much on the length of exposure this idea appears reasonable. Asbestos is extremely insoluble.

As intercellular fluid is saline in nature the importance of fibre solubility in saline solution has long been recognised. If fibres are soluble in physiological saline solution then, provided the dissolved components are not toxic, the fibres

should be safer than fibres which are not so soluble. The shorter the time a fibre is resident in the body the less damage it can do. H. Förster in The behaviour of mineral fibres in physiological solutions' (*Proceedings of 1982 WHO IARC* Conterence. Copenhagen. Volume 2. pages 27-55(1988)) discussed the behaviour of commercially produced mineral fibres in physiological saline solutions. Fibres of widely varying solubility were discussed.

International Patent Application No. WO87/05007 disclosed that fibres comprising magnesia. silica. calcia and less than 10 wt% alumina are soluble in saline solution. The solubilities of the fibres disclosed were in terms of parts per million of silicon (extracted from the silica containing material of the fibre) present in a saline solution after 5 hours of exposure. The highest value revealed in the examples had a silicon level of 67 ppm. In contrast, and adjusted to the same regime of measurement, the highest level disclosed in the Förster paper was equivalent to approximately 1 ppm. Conversely if the highest value revealed in the International Patent Application was converted to the same measurement regime as the Förster paper it would have an extraction rate of 901,500 mg Si/kg fibre - i.e. some 69 times higher than any of the fibres Förster tested, and the fibres that had the highest extraction rate in the Förster test were glass fibres which had high alkali contents and so would have a low melting point. This is convincingly better performance even taking into account factors such as differences in test solutions and duration of experiment.

International Patent Application No. WO89/12032 disclosed additional fibres soluble in saline solution and discusses some of the constituents that may be present in such fibres. Among such constituents are ZrO<sub>2</sub> and this document claims (among other things) processes using fibres of composition (in weight %):- ZrO<sub>2</sub> 0.06-10%: SiO<sub>2</sub> 35-70%: MgO 0-50%; CaO 0-64.5%. However the patent actually discloses a much more limited range of zirconia containing materials and these are listed in Table 1 below ranked on silica content. None of the disclosed zirconia containing compositions were tested for shrinkage and hence usefulness in high temperature applications; all that these fibres were tested for was ability to withstand a fire test and Table 1 indicates that the results of this test were not very predictable; there does appear to be a trend with silica content but no trend is discernible with zirconia content.

European Patent Application No. 0399320 disclosed glass fibres having a high physiological solubility.

Further patent specifications disclosing selection of fibres for their saline solubility are European 0412878 and 0459897. French 2662687 and 2662688. PCT WO86/04807 and WO90/02713.

Table 1

Test	SiO <sub>2</sub>	CaO	MgO	Al <sub>2</sub> O <sub>3</sub>	ZrO <sub>2</sub>	Fire	SiO.			Al <sub>2</sub> O,	ZrO <sub>2</sub>
	wt%	wt%	wt%	wt%	wt%	1		mol%	mol%	mol%	mol%
						Pass/ Fail	<b>1</b> :				
			· 	:				127.00	D 40	0.5	0.1
174	63.5	35.55		0.88	0.21		61.83	<del> </del>		<del></del>	
178	60	38.3	0.48	0.36	0.54		<del></del>	140.14		0.21	0.26
177	59.7	38.7	0.46	0.34	0.50	<u> </u>		140.53		0.20	0.24
176	59.5	39.1	0.42	0.31	0.42	-	58.1	40.91	0.61	0.18	0.2
182a	59.4	34.9	2.06	0.38	2.31	P	58.69	36.94	3.03	0.22	1.11
181	59.2	36.6	1.13	0.32	0.83	Р	58.8	38.94	1.67	0.19	0.4
179	59.2	37	0.98	0.35	0.58	P	58.74	39.33	1.45	0.2	0.28
175	59.2	39.1	0.41	0.33	0.4	P	57.99	41.03	0.6	0.19	0.19
183	59.05	34.84	3.08	0.3	2.65	P	57.65	36.44	4.48	0.17	1.26
186	59.05	36.94	2.57	0.38	3.27	P	56.63	37.95	3.67	0.21	1.53
191	58.6	33.5	2.72	0.58	3.67	P	58.21	35.65	4.03	0.34	1.78
192	58.4	33.2	2.59	0.65	3.69	l P	58.39	35.56	3.86	0.38	1.8
- 189	58.19	35.39	3.26	0.39	3.36	-	56.59	36.87	4.73	0.22	1.59
184	57.96	35.17	3.55	0.42	3.11	F	56.44	36.69	5.15	0.24	1.48
190	57.86	35.66	3.22	0.36	3.37	F	56.33	37.19	4.67	0.21	1.6
185	57.8	34.4	3.74	0.56	3.12	F	56.62	36.1	5.46	0.32	1.49
188	57.7	36	3	0.2	3.3	Р	56.31	37.64	4.36	0.12	1.57
187	56.88	36.45	4	0.32	3.3	-	54.86	37.66	5.75	0.18	1.55
193	56.65	31.9	3.35	3.35	4.5	F	56.66	34.18	4.99	1.97	2.19
180	54.3	32.75	10.2	1.29	0.58	F	51.41	33.22	14.39	0.72	0.27
182	46.85	29.2	20.6	2.03	0.84	F	42.42	28.33	27.8	1.08	0.37

The refractoriness of the fibres disclosed in these various prior art documents varies considerably. The maximum service temperature of any of the above mentioned fibres (when used as refractory insulation) is up to 815°C (1500°F).

Among saline soluble commercial fibres usable at temperatures higher than 815°C are SUPERWOOL TM a fibre manufactured by The Morgan Crucible Company plc and which has a maximum use temperature of 1050°C and a composition of SiO<sub>2</sub> 65wt%: CaO 29wt%: MgO 5wt%: Al<sub>2</sub>O<sub>3</sub> 1wt%. A similar fibre is INSULFRAX TM a fibre made by Carborundum Company which has a continuous use limit of 1000°C (1832°F) and which melts at 1260°C (2300°F). This has a composition of SiO<sub>2</sub> 65wt%: CaO 31.1wt%: MgO 3.2wt%: Al<sub>2</sub>O<sub>3</sub> 0.3wt% and Fe<sub>2</sub>O<sub>3</sub> 0.3wt%.

Use of ZrO<sub>2</sub> as a constituent in aluminosilicate fibres to provide high temperature resistance is known (see European 0144349). However it is by no means apparent that this effect is transferable to saline soluble fibres and the disclosure of International Patent Application No. WO89/12032 discussed above would tend to suggest that it is not.

The applicant's earlier International Patent Application WO93/15028 (from which this application claims priority) disclosed saline soluble fibres usable at temperatures in excess of 1000°C but gave no indication that fibres could be used at still higher temperatures. The applicants have found that some of the fibres disclosed in WO93/15028 (e.g. fibre A2-13 from Table 9 of WO93/15028) are in fact usable at temperatures of up to 1260°C and even higher. In general the applicants have found that fibres of specified compositions (including zirconia containing fibres) are usable at temperatures up to and beyond 1260°C. The applicants have realised that failure of fibres at high temperature occurs primarily upon devitrification of the fibre: if on devitrification insufficient silica is left the fibres will fail through having a shrinkage of greater than 3.5%. Accordingly the applicants have looked to what materials are formed on devitrification.

In the following where reference is made to a saline soluble fibre this is to be taken as meaning a fibre having a total solubility of greater than 10ppm in saline solution as measured by the method described below, and preferably having much higher solubility.

Figure 1 shows a three axis composition diagram for the constituents CaO. MgO. and ZrO<sub>2</sub>: this diagram omits all other constituents so that the sum of CaO, MgO, and ZrO<sub>2</sub> at all points is 100%. Silica is in excess at all points as described below.

For fibres where CaO > MgO + 2ZrO<sub>2</sub> all of the MgO is bound as CaO.MgO.2SiO<sub>2</sub>; all of the ZrO<sub>2</sub> is bound as 2CaO.ZrO<sub>2</sub>.4SiO<sub>2</sub>; and any excess

CaO is bound as CaSiO<sub>3</sub>. These fibres lie in region 1 of Figure 1 and in the following are referred to as excess CaO fibres.

For fibres where MgO > CaO all of the CaO is bound as CaO.MgO.2SiO<sub>2</sub>; all of the ZrO<sub>2</sub> is bound as ZrO<sub>2</sub>.SiO<sub>2</sub>; and the excess MgO is bound as MgO.SiO<sub>2</sub>. These fibres lie in region 2 of Figure 1 and in the following are referred to as excess MgO fibres.

For the fibres in region 3 of Figure 1 where CaO > MgO and CaO < MgO + 2ZrO<sub>2</sub>, all of the MgO is bound as CaO.MgO.2SiO<sub>2</sub>; the rest of the CaO is bound as 2CaO.ZrO<sub>2</sub>.4SiO<sub>2</sub>; and the excess ZrO<sub>2</sub> is bound as ZrO<sub>2</sub>.SiO<sub>2</sub>. These fibres are referred to in the following as excess ZrO<sub>2</sub> fibres.

The applicants have defined a term "SiO<sub>2</sub> excess" which indicates the amount of silica left once the above mentioned constituents (CaO, MgO, and ZrO<sub>2</sub>) have crystallised. The value of SiO<sub>2</sub> excess is calculated by subtracting from the total quantity of silica present that amount that should crystallise as silicates with the other constituents CaO, MgO, and ZrO<sub>2</sub> assuming all of the CaO, MgO, and ZrO<sub>2</sub> crystallise as the materials mentioned above. In most of the compositions studied alumina is present to some extent and so the applicants also assume that alumina crystallises as Al<sub>2</sub>O<sub>3</sub>.SiO<sub>2</sub> and to calculate SiO<sub>2</sub> excess this quantity is subtracted also. Only the above named constituents are used in calculating the SiO<sub>2</sub> excess as other chemical constituents are present in only small amounts. For other chemical constituents similar considerations apply. It has been found by the applicants that when the SiO<sub>2</sub> excess is greater than 21.8mol% the fibres tend to have a resistance to temperature of up to 1260°C

The applicants have found that for the excess CaO compositions the situation is complicated by a eutectic formed between the two crystalline materials diopside (CaO.MgO.2SiO2) and wollastonite (CaSiO<sub>3</sub>) that has a damaging effect on high temperature resistance. Thus the present invention excludes those excess CaO compositions that have a calculated diopside to wollastonite ratio in the range 1.8 to 5.25.

The physical basis for the importance of SiO<sub>2</sub> excess may be that it indicates how much silica is left to maintain a glassy phase on crystallisation of the other constituents as silicate materials. Further, the silicate materials that form on devitrification may become liquid or flow at 1260°C so causing shrinkage.

The quantity of potentially fluxing constituents such as alkali metals and other incidental impurities (e.g. iron oxides) should be kept low.

Accordingly the present invention provides a refractory fibre for which a vacuum cast preform of the fibre has a shrinkage of 3.5% or less when exposed to 1260°C for 24 hours and comprising CaO, SiO<sub>2</sub>, MgO, optionally ZrO<sub>2</sub> and/or less than 0.75mol% Al<sub>2</sub>O<sub>3</sub>, any incidental impurities amounting to less than 2mol% in total, and in which the SiO<sub>2</sub> excess (defined as the amount of SiO<sub>2</sub> calculated as remaining after the above named constituents are crystallised as silicates) exceeds 21.8mol%, with the proviso that, if the amount of CaO is greater than the sum of the amount of MgO and twice the amount of ZrO<sub>2</sub> the calculated ratio of diopside to wollastonite does not lie in the range 1.8 to 5.25.

The applicants have also found that for those fibres that have a satisfactory shrinkage at 1260°C the saline solubility of the fibres produced appears to increase with increasing amount of MgO present whereas ZrO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> are detrimental to solubility. The invention therefore also provides preferred saline soluble fibres of the composition specified above and in which the MgO excess [defined as MgO - (ZrO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub>)] is greater than 10mol%, such fibres tending to have a total solubility of MgO + SiO<sub>2</sub> + CaO of greater than 50ppm (see below for measurement details). More preferably the MgO excess is greater than 11.2mol% such fibres tending to have extremely high solubility of about 100ppm or more. Yet more advantageously, so far as solubility is concerned, the MgO excess is greater than 15.25mol%; all of the fibres measured having an MgO excess greater than 15.25mol% had solubilities in excess of 100ppm.

As a consequence of inventing these fibres the invention also provides a saline soluble fibre characterised in that a vacuum cast preform of the fibre has a shrinkage of 3.5% or less when exposed to 1260°C for 24 hours.

The applicants have investigated, for their saline solubility and refractoriness, a range of compositions based on CaO/MgO/SiO<sub>2</sub> fibres with additional constituents Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, and TiO<sub>2</sub>. These fibres were formed by blowing the molten constituents from a melt stream in a conventional manner but the invention is not limited to blown fibres and also encompasses fibres formed by spinning or any other means.

Tables 2 & 3 show the results of these tests. Table 2 indicates for each the linear shrinkages at 800, 1000, 1200, and 1260°C (not all samples measured at every temperature); weight percent composition: mole percent composition (based on the constituents CaO, MgO, SiO, Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, and TiO<sub>2</sub>); SiO<sub>2</sub> excess

(as defined above) and, for the CaO excess fibres, the calculated diopside to wollastonite ratio. Table 3 indicates for each the weight percent composition; mole percent composition (based on the constituents CaO, MgO, SiO<sub>2</sub> Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, and TiO<sub>2</sub>); solubilities of various constituents; and MgO excess (as defined above) Each sample that has a satisfactory shrinkage of 3.5% or less at 1260°C is indicated by a composition shown in bold. Those compositions that fail to meet the shrinkage criterion are indicated in italics. Other compositions are shown falling within the described ranges but for which the high temperature shrinkage was not measured; these compositions are indicated in plain text. Those compositions where a fibre could not be made or where the fibre was of too poor a quality for the solubility to be measured are indicated with X's.

A pattern emerges which is described below with reference to Table 2.

The fibres above and including line A all have a SiO<sub>2</sub> excess of less than 21.8mol% and all (where measured) fail the shrinkage criterion that a vacuum cast preform of the fibre has a shrinkage of less than 3.5% when exposed to 1260°C for 24 hours.

The fibres above and including line B and below line A all have a TiO<sub>2</sub> content of greater than 1.25mol% and all fail the shrinkage criterion.

The fibres above and including line C and below line B all have a Al<sub>2</sub>O<sub>3</sub> content of greater than 0.75mol% and all fail the shrinkage criterion.

The fibres below line C are grouped according to their relative amounts of CaO, MgO, and ZrO, (i.e. as to their positions in Figure 1)

The fibres above and including line D and below line C are the excess MgO fibres (region 2 of Figure 1) and are sorted on SiO<sub>2</sub> excess.

The fibres above and including line E and below line D are the excess ZrO, fibres (region 3 of Figure 1) and are sorted on SiO<sub>2</sub> excess.

The fibres below line E are the excess CaO fibres and are sorted on the diopside to wollastonite ratio.

The fibres above and including line F and below line E are excess CaO fibre for which the diopside to wollastonite ratio is greater than 5.25.

The fibres above and including line G and below line F are excess CaO fibre for which the diopside to wollastonite ratio is less than 5.25 but greater than 1.8.

The fibres below line G are excess CaO fibre for which the diopside to wollastonite ratio is less than 1.8.

Looking first to the excess MgO fibres most pass the shrinkage criterion at 1260°C (where tested). B7D. BZ-440C, B7C, and BZ-4150C all contain relatively high levels of Fe<sub>2</sub>O<sub>3</sub> (1.1wt% for B7D and 0.6wt% for the others).

D3 and D8 contain relatively high levels (0.71mol% and 0.74 mol%) of TiO<sub>2</sub> and it may be that this, in combination with other impurities, has led to tailure. It should be noted that D9 has 0.65mol% TiO<sub>2</sub> and has a satisfactory shrinkage.

BZ-440A, B7A, BZ-4150A, and BZ-560B have varying amounts of Na<sub>2</sub>O present (0.3-1.0wt%) and this may contribute to their failure.

BZ-4150B has a Al<sub>2</sub>O<sub>3</sub> content of 0.64mol% and fails the shrinkage criterion. This should be contrasted with BZ-4150 which has a similar composition but with only 0.06mol% Al<sub>2</sub>O<sub>3</sub> and which passes the shrinkage criterion. In further contract BZ-560E has an alumina content of 0.62mol% and passes the shrinkage criterion: this composition has a much higher ZrO<sub>2</sub> content than BZ-4150B and the applicant believe that the presence of ZrO<sub>2</sub> allows the fibres to tolerate much higher levels of impurities than would otherwise be the case.

D3 only just fails with a shrinkage of 3.8% and B19 only has a shrinkage of 3.6% at 1260°C and both may in fact be errors in measurement.

Looking next to the excess ZrO<sub>2</sub> fibres all apart from BZ-407, BZ-429 and BZ-430 pass the shrinkage criteria at 1260°C (where tested). These results may indicate that the incidental impurities (shown as "Others" in Table 2) are having an effect as BZ-429 and BZ-430 show high levels of impurities (1.1 and 0.9 wt% respectively) that on analysis included 0.4 and 0.3 wt% respectively of Na<sub>2</sub>O. BZ-430 only just failed the shrinkage criterion (3.7% shrinkage) and this may be due to error in measurement.

Turning now to the excess CaO fibres the pattern is clear but not exact. Fibres that have a diopside to wollastonite ratio of between 5.25 and 1.8 fail the

shrinkage criterion. Those with a diopside to wollastonite ratio outside this range tend to pass. The fit is not exact and the fibres that fail to meet the shrinkage criterion are the following.

Among the excess CaO fibres with a diopside to wollastonite ratio in excess of 5.25 those that fail the shrinkage criterion include BZ-418, and BZ-29 which have low enough shrinkages that they may be the result of experimental error and these fibres may in fact have a satisfactory shrinkage.

BZ-421. B13, BZ-422. BZ-417, and BZ-416 also fail and although initial indicators were that this had something to do with the level of CaO this now appears to be incorrect. The failure to meet the shrinkage criterion may be due to the presence of fluxing constituents or otherwise. A possible reason for failure of BZ-29 and BZ-421 may be their high Al<sub>2</sub>O<sub>3</sub> content (0.55 and 0.51 mol% respectively) acting alone or in combination with impurities.

For the excess CaO fibres having a diopside to wollastonite ratio of less than 1.8 the only fibre proven to fail was fibre E24 which although passing a 1260°C test failed a 1200°C test. This result may have been due to experimental error, fluxing components, or otherwise.

Table 3 shows the solubilities of the fibres shown in Table 2 but ranked on MgO excess. Although by no means exact it can be seen that there is a trend in total solubility that closely follows MgO excess.

In any event the trend appears to be that excess CaO fibres perform poorly (perhaps due to the formation of CaSiO<sub>3</sub> which is not formed in excess MgO or excess ZrO<sub>2</sub> fibres) whereas excess MgO and excess ZrO<sub>2</sub> fibres perform better. Taken to the extreme this would indicate that a high MgO, low CaO, low ZrO<sub>2</sub>, low Al<sub>2</sub>O<sub>3</sub> fibre would have very high solubility and low shrinkage. However the applicant's experience is that such fibres are difficult to form (see Compositions A2-33, A2-32, A2-28). Equally fibres that are too high in SiO<sub>2</sub> are difficult or impossible to form. The exact boundaries are difficult to ascertain and this invention only encompasses fibres that meet the above stated shrinkage requirements.

The applicants have tested some fibres to higher temperatures.

Fibres BZ-400, BZ-440, BZ-48, and BZ-54 were tested to 1350°C and all failed having shrinkages in excess of 20%.

Fibres BZ-400. BZ-36. BZ-46. and BZ-61 were tested to 1300°C and had shrinkages, respectively. of 6.2%. 17.9%. 19.6%. and 3.1%. BZ-61 is in the excess MgO region and the applicants surmise (since 2CaO.ZrO.4SiO. is not formed in this region) that it is this constituent that causes failure at 1300°C.

The fact that fibre shrinkage is so dependent on temperature (the fibres failing over such short temperature ranges as 1260°C to 1300°C and 1300°C to 1350°C) is a clue as to how experimental errors may arise. In a typical experimental furnace running at a nominal 1260°C temperatures can easily range from 1250°C to 1270°C both physically (from front to back or centre to wall of furnace) and in time (as the furnace controller supplies or stops current to the furnace). A 20°C temperature difference could easily move a sample from a temperature at which it passes to one at which it fails the 3.5% shrinkage criterion. As mentioned above this may explain the shrinkages of just over 3.5% found for compositions B19. D3. BZ-430. BZ-418 and BZ-29.

During the shrinkage tests some of the sample preforms used were also inspected to ascertain whether they reacted adversely with the ceramic boards (alumina or mullite boards) on which they rested during the test. It was found that the excess CaO fibres with a diopside to wollastonite ratio of less than 1.8 reacted particularly badly with mullite boards and further that due to acciular crystal growth the fibres tended to lose strength.

The following describes in detail the methods used to measure shrinkage and solubility.

Shrinkage was measured by proposed ISO standard ISO/TC33/SC2/N220 (equivalent to British Standard BS 1920, part 6.1986) with some modifications to account for small sample size. The method in summary comprises the manufacture of vacuum cast preforms, using 75g of fibre in 500cm³ of 0.2% starch solution, into a 120 x 65mm tool. Platinum pins (approximately 0.1-0.3mm diameter) were placed 100 x 45mm apart in the 4 corners. The longest lengths (L1 & L2) and the diagonals (L3 & L4) were measured to an accuracy of ±5µm using a travelling microscope. The samples were placed in a furnace and ramped to a temperature 50°C below the test temperature at 400°C/hour and ramped at 120°C/hour for the last 50°C to test temperature and left for 24 hours. The shrinkage values are given as an average of the 4 measurements.

It should be noted that although this is a standard way of measuring shrinkage of fibre it has an inherent variability in that the finished density of the

preform may vary depending on casting conditions. Further it should be noted that fibre blanket will usually have a higher shrinkage than a preform made of the same fibre. Accordingly the 3.5% figure mentioned in this specification is likely to translate as a higher shrinkage in finished blanket.

The applicants have looked to the various incidental impurities that can occur in inorganic oxide refractory fibres (e.g. alkali oxides and iron oxide) and have found that the impurity levels that can be tolerated vary according to the proportions of the main constituents of the fibre. Fibres containing high levels of ZrO<sub>2</sub> for example can tolerate higher levels of Na<sub>2</sub>O or Fe<sub>2</sub>O<sub>3</sub> than fibres with low levels of ZrO<sub>2</sub>. Accordingly the applicants propose a maximum level of incidental impurities of 2mol%, the maximum level that will be tolerable will however vary as mentioned above.

Solubility was measured by the following method.

The fibre was first chopped - 2.5 g of fibre (deshotted by hand) was liquidised with 250 cm<sup>3</sup> of distilled water in a domestic Moulinex (Trade Mark) food blender for 20 seconds. The suspension was then transferred to a 500 cm<sup>3</sup> plastic beaker and allowed to settle after which as much liquid as possible was decanted and the remaining liquid removed by drying in an oven at 110°C.

The solubility test apparatus comprised a shaking incubator water bath, and the test solution had the following composition:-

Compound	Name	<u>Grams</u>
NaCl	Sodium chloride	6.780
NH,Cl	Ammonium chloride	0.540
NaHCO <sub>3</sub>	Sodium bicarbonate	2.270
Na <sub>2</sub> HPO <sub>4</sub> .H <sub>2</sub> O	Disodium hydrogen phosphate	0.170
Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub> .2H <sub>2</sub> O	Sodium citrate dihvdrate	0.060
H <sub>2</sub> NCH <sub>2</sub> CO <sub>2</sub> H H <sub>2</sub> SO <sub>1</sub> s.g. 1.84	Glycine Sulphuric acid	0.450 · 0.050

The above materials were diluted to 1 litre with distilled water to form a physiological-like saline solution.

 $0.500 \text{ grams} \pm 0.0003 \text{ grams}$  of chopped fibre was weighed into a plastic centrifuge tube and 25 cm<sup>3</sup> of the above saline solution added. The fibre and saline solution was shaken well and inserted into the shaking incubator water bath maintained at body temperature (37°C = 1°C). The shaker speed was set at 20 cycles/minute.

After the desired period (usually 5 hours or 24 hours) the centrifuge tube was removed and centrifuged at 4500 revs/minute for approximately 5 minutes. Supernatant liquid was then drawn off using a syringe and hypodermic needle. The needle was then removed from the syringe, air expelled from the syringe, and the liquid passed through a filter (0.45 micron cellulose nitrate membrane filter paper [WCN type from Whatman Labsales Limited]) into a clean plastic bottle. The liquid was then analysed by atomic absorption using a Thermo Jarrell Ash Smith - Hiefje II machine.

The operating conditions were as follows using a nitrous oxide and acetylene flame:-

ELEMENT	WAVELENGTH (nm)	BAND <u>WIDTH</u>	CURRENT_ (mA)	FLAME
Al	309.3	1.0	8	Fuel Rich
SiO <sub>2</sub>	251.6	0.3	. 12	Fuel Rich
CaO	422.7	1.0	7	Fuel Lean
MgO	285.2	1.0	3 .	Fuel Lean

The procedure and standards adopted for determining the above elements were as set out below.

SiO<sub>2</sub> can be determined without dilution up to 250 ppm concentration (1 ppm lmg/Litre). Above this concentration an appropriate dilution was made volumetrically. A 0.1% KCl solution (0.1g in 100 cm<sup>3</sup>) was added to the final dilution to prevent ionic interference. NB If glass apparatus is used, prompt analysis is necessary.

From a stock solution of 1000 ppm pure ignited silica (99.999%) (fused with Na<sub>2</sub>CO<sub>3</sub> at 1200°C for 20 minutes in a plattium crucible (0.2500g SiO<sub>2</sub>/2g Na<sub>2</sub>CO<sub>3</sub>) and dissolved in dilute hydrochloric acid (4 molar) made up to 250cm<sup>3</sup> with distilled water in a plastic volumetric flask) the following standards were produced:-

STANDARD (ppm SiO <sub>2</sub> )	STOCK SOLUTION (cm <sup>3</sup> )
10.0	1.0
20.0	2.0
30.0	3.0
50.0	5.0
100.0	10.0
250.0	25.0

Add 0.1% KCl to each standard before making to 100cm<sup>3</sup>.

Aluminium may be measured directly from the sample without dilution. Standards of 1.0, 5.0 and 10.0 ppm Al may be used. For calibration readings are multiplied by 1.8895 to convert from Al to Al<sub>2</sub>O<sub>3</sub>.

A standard Al atomic absorption solution (e.g. BDH 1000 ppm Al) was bought and diluted using an accurate pipette to the desired concentration. 0.1% KCl was added to prevent ionic interference.

Calcium may require dilutions on the sample before determination can be carried out (i.e. x 10 and x 20 dilutions). Dilutions must contain 0.1% KCl.

A standard Ca atomic absorption solution (e.g. BDH 1000 ppm Ca) was diluted with distilled water and an accurate pipette to give standards of 0.5, 4.0 and 10.0 ppm. 0.1% KCl is added to prevent ionic interference. To convert readings obtained from Ca to CaO a factor of 1.4 was used.

Magnesium may require dilutions on the sample before determinations can be made (i.e. x 10 and x 20). Add 0.1% KCl to each dilution. To convert Mg to MgO multiply by 1.658.

A standard Mg atomic absorption solution (e.g. BDH 1000 ppm Mg) was diluted with distilled water and an accurate pipette to give standards of 0.5, 1.0 and 10.0 ppm Mg. 0.1% KCl was added to prevent ionic interference.

All stock solutions were stored in plastic bottles.

The above has discussed resistance to shrinkage of preforms exposed to 1260°C for 24 hours. This is an indication of the maximum use temperature of a fibre. In practice fibres are quoted for a maximum continuous use temperature and a higher maximum exposure temperature. It is usual in industry when

selecting a fibre for use at a given temperature to choose a fibre having a higher continuous use temperature than that nominally required for the intended use. This is so that any accidental increase in temperature does not damage the fibres. It is quite usual for a margin of 100 to 150°C to be given. Accordingly this invention extends to use of the claimed fibres at elevated temperatures (i.e. at temperatures where the refractoriness of fibres is important) and not just to use at 1260°C.

In selecting a fibre a balance has to be struck between refractoriness of the fibre and saline solubility of the fibre. For example the applicants have found the best high solubility fibre (total solubility greater than 100ppm) is probably composition B7 as that has a shrinkage of 2.7% at 1260°C. In contrast the best refractory fibre is probably BZ-560 which has a shrinkage of only 2.1% at 1260°C but has a total solubility of only 27ppm. Although there are other fibres with a lower shrinkage this fibre also has the property of retaining in large part its resilience on firing to 1260°C - many of the fibres become rigid after firing due to crystallisation and sintering. It appears that high levels of ZrO<sub>2</sub> help to overcome this (BZ-560 has 7.64mol% ZrO<sub>2</sub>) but at the same time reduce solubility.

It will be evident from the above that incidental impurity levels are preferably kept as low as possible. The applicants surmise that as the various crystalline materials crystallise from the fibres impurities migrate to the grain boundaries and concentrate there. Thus a small impurity can have a very large effect.

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CaO M	21.05	37.55	20.06	35.10	21 59	<u> </u>	23.74	25.02	20.02	27.30	1.035	16.26		1.82	1.1.8.2 2.5.8.8	11.82 25.83 17.11	13.83 17.11 3.83	11.82 13.83 17.11 18.20 26.57	11.82 12.83 17.12 13.03 35.03	11.82 17.11 17.11 26.57 27.07	25.8% 17.11 17.11 26.57 27.07 27.18	1.82 12.83 17.11 17.11 26.57 33.08 27.07 15.09	25.8% 25.8% 26.57 25.07 27.00 27.00 15.00 12.76	25.8% 25.8% 17.11 2.6.57 27.07 27.07 15.09 12.76 17.71	11.82 18.83 18.92 18.92 19.05	23.8 % 11	13.8% 13.8% 13.8% 13.8% 20.57 22.0% 12.0% 12.7% 11.7%	11.82 13.88 13.88 13.88 13.00 12.00 12.76 13.00 18.57 16.49	13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83 13.83	13.83 13.83 13.83 13.83 13.00 12.76 13.00 13.00 13.00 13.00 13.00 13.00 13.00 13.00 13.00 13.00 13.00 13.00 13.00 13.00	13.83 13.83 13.83 13.83 13.00 12.76 13.00 10.00 10.00 10.00 10.00 10.00 10.00	13.83 12.11 13.83 13.00 12.00 12.00 12.70 12.70 12.70 12.70 12.70 12.70 13.71 18.92 18.93 18.93 18.45	13.83 13.12 13.13 13.00 12.70 12.70 12.70 12.70 12.70 12.70 13.71 18.92 18.93	13.83 13.11 13.11 13.00 13.70	13.83 13.83 13.83 13.00 12.70 12.70 12.70 12.70 12.70 12.70 12.70 12.70 13.71 18.92 18.53 18.45
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Composition MgO ZA12	18.31	19 29	17.38	65.81	22.95	16.82	15 16	18.24	20.65	16.85	17.44	16.91	6.87	17.66	11 05	36.31	11.47	. 06 92	11.00	23.30	15.12:	17.75	30 23 "	1111	14.05	18.76	14.12;	13.92.	17.07	15.03	27 03	14.51	18 09	91 91	15.19	26 70	20 55	22 88	14.56	11.73
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Others	L				2				9	_					_					_		_	_		_					-		_	_			6	<u> </u>			
SiCr2	5	9.00	63.9	66 2	65.7	64.2	61.5	67.8	9.89	645	65.7	65.3	9	1 65.7	201	 •	69.1	GB 0.5	_	61 80	63.8	68.33	71.24	62.3			62.5			63.7	=	65.6	69.2	67.3	63.	73 09	70 43	71 +8	67.4	67.8
rio2	9		0.1	_	- 0	70			<b>6</b> .1	0.7		=	_				9		<u>-</u>					0	0.0	<u>-</u>	<u>-</u>	<u>-</u>	6.0											
wr"• Al2O3 TiO2	5	10	10	+ 0	20	11	0.2	0.3	0.7	0.2	0.2	0.1	0.7	0.1	0.25	<u>0</u>	-C	67.0	_	-	0.3		0.19		=	£.	0.	=	=	-6-	17.0	T.	0.07	2	=	0.23	0.15	0.33	=	0.7
_ CI	53	0.2	. 7.5		0 7	1.1	15.8	8.0	0.4	7.2	5.2	7.7	50 47.	9	Ξ.	17.6	=	186'0	15.1	16.9	13.6		1.23	13.1	15.6	10.2	5	12.	1.87	15.7	<u>5</u>	=	1.13	5.7	18.2	0.36	1.58	(1.83	9.8	10.8
Composition MgO /rC	12.6	13.5	611	13.2	16.5	11.5	6.6	13.1	¥2	11.5	12.1	11.7	11.6	12.2	22 85	y.9	5.5	19.26	6	16.57	2	126	22.31	2.0	9.1	9.0	9.1	6	~	8.6	3.0	9.1	12.76	11.3	9.7	9 61	14.52	16.34	8.6	6.6
	16.2	8:	155	60/	657	133	12.4	17.9	<b>80</b>	133	15.9	15.2	<u>+:-</u>	2	<u> </u>	<b>E S</b>	17.7	8 12.	11.9	H 58	12.5	17.45	9 <del>+</del> +	13.	11.7	=	11.7	11.7	6.2	:- -0 -	6.77	13.2	15.17	13.6	 	6.36	11.58	9.36	13.1	=
209	4.8	3.9	2	j.	Ç.	٠٠ ن	7.3	7.7	2.5	9.4	2.1	3.2	M.	7		<u>8.</u>	3.2		J. 5.		2.1.			14	7:	7.6	3.1	7.	<u></u>	2.7	•••	~	. –	٠ ا	1.7				2.1	2.7
C - 15	-				_			2.8	2.7		2.3	2.7	2.3			1.										2.8		<u>×</u>				7							-	_
Shimkage 1000C 1200C 1260C   CaO	-			7	5 /	_		<u></u>	1.2		<u> </u>	6.1	Ξ	6.9		<u>-</u>	<u>-</u>	- 2		=	=	1.7	<b>80</b>	_		9.1		Ξ	=	= :2	7.	-	5.1	8.0	7		<u></u>	Ξ	8.6	<del>-</del> .
Shri 800C 100	-		-	-	=		 8.0	1.2	<b>-</b>	_	0.7		8.0	0.7	-	0.3	0.9	1.5	_	=	0.7	=				_	• •			-	<u>-</u> -	0.2	_	6.5	-0.				9.3	7
	. )();		. ×ić.			87.4150B				150.1	- -	150		137	<u> </u>	7		ء	DZ-560E	_ _	30		<u> </u>	BZ-560B	.2095-ZE	919	1095-ZE	260	<u>.</u>	9,	<i>ي</i>	- 19	_		09	7	11	72	63	
స	B7-410(	1876	BZ-1151K	ă:	6/8	87.1	BZ-20	134	<u>=</u>	82-1150	BZ-140	85-4150	20	BZ-437	A2-33	18.28	B17	A2-20	DZ-:	A2-20	85-28	759	A2-20	87-5	18.2	182,610	BZ.	HZ-560	60	BZ-56	A2-27	BZ-	A2-6	2	BZ-60	A2-32	A2-17	A2-22	102-63	BZB

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Sit)2 Diopside  Excess Wolfastonite Ratio											,																							-		_		7
SiO2 Excess	37.75	42.65	07.9	22.50	22.51	22.90	23.65	24.35	2.136	24.76	25.21	25 53	25 64	25.78	26 22	26 22	26.33	26.40	26.71	26.85	27.04	27.60	28 53	28 98	29.21	29.40	45.62							_	┙			31.47
SiU2	88 89	71.32	0 S	02.30	K1 18	62.72	62.15	62.33	63.12	63.90	63.58	65.67	63.26	61.12	63.34	63.21	64.11	63.86	63.47	65.75	64.43	63.85	65.21	65.03	65.33	65.54	2.42	65.80	. 66.02	67.34	66.24	99.99	68.88	67.16	68.91	65.76	64.63	66.54
Tio2 s			1													0.07															0.07							•
mol°e Al2O3 T	90.0	0.11		0.12	910	0.12	0.12	90.0	0 12	0.12	0.12	0.12	90.0	6.17	0.35	0.57	0.1	0.12	<u></u>	0.19	0.E	0.1	0.12	6.29	0.06	2.0	7 0	0,00	0.06	0.13	0.11	0.17	0.18	- O	0.0	0.12	0.12	0
7	0.58	90.1		71.7	7 93	3.53	3.20	09.9	7 7	3.17	3.53	1.00	5.15	1.57	2.52	143	3.23	<del>1</del> .19	9+ 1	9.06	2.76	2.56	2.58	3.34	3.15	2.19	97.0	2.73	2.57	9.15	2.11	3.38	5.36	1.70	7.7	6.19	7.7	1.61
Composition MgO 2rt)	17.55	14.61	7,7%	77.77	8 02	14.27	16.36	15.18	14 47	13.37	11.43	7.80	14.81	17.1	16.44	17.07	14.38	14.60	17.11	7.85	14.53	16.63	14.16	17.58	11.28	14.39	1 1 1	1 5	11.53	7.99	14.31	14.16	7.65	1.13	12.58	7.75	13.76	3
Cac Mg	<u> </u>	12.90	80.2	10.03	19 6R	19.36	18.06	15.82	13 19	19.41	18.31	19.41	16.59	19.03	17.36	17.63	18.16	17.24	17.57	17.15	18.17	16.84	17.94	16.76	17.18	17.76	2 2	16.97	16.82	15.39	17.04	15.69	17.93	16.90	15.74	20.18	19.05	1.69
Others	$\mid$		1	5 -	. 0	7.0	7.0	0.5	6.0	7.0	0.1	9.0	6.0	9.5	0.7	T:0	7.0	9.0	<del>1</del> .0	0.7	<del>-</del>	6.0	-0	-	<del>-</del>	<del>-</del> (	٥ <u>٠</u>	, v		9.0	0.3	7.0	0.5	0.3	2	0.5	+.0	0.3
SiO2 Or	72.25	73.43		63.5	8.09	64.2	63.5	61.2	63.2	64.8	68	62.7	67.9	65.7	64.9	65.7	65.8	63.9	8.89	61.2	66.1	65.8	9.99	8.99	8.99	67.3	64.9	6.5.6	67.7	61.8	68.3	67.3	67.1	69.4	69.4	63.2	8.8	68.5
•			7	_								_				0.0														_	0.7				1			<del>-</del>
мт°. Aizo3 Tio2	0.11	61.0		7.0		0.7	0.2	0.0	0 2	0.3	0.7	0.2	0.1	0.3	9.0	_	0.7	0.7	0.7	0.3	0.7	0.7	0.7	0.5	-	7.0	7.0			0.7	0.7	0.3	0.3	0.7	5	0.7	0.7	0.7
-	l		.		7	7.4	6.7	3.3	8.5	9.9	7.4 :	13.7	10.7	5.4	5.3	3.1.	8.9	9.8	3.1	7.3	5.8	7.		_	9.9	9 ,	0.7			17.5	1.7	•	10.7	3.6	5.6	12.2	<b>-</b>	7.
Composition MgO ZrO2	35	8 :	2];	20			1.2	_	9.7	1.6				9.7	13	6.1	6.6				0	1.5	9.1	2	80.	6.6	n :	, ,	; =	100	6.6	9.6	40	8.6	8.5	S	7.6	9.7
	I_	12.4 10.	70.7	2 2	17.1	8.8	17.2	14.5	11	18.4	17.5	17.3	15.4	8.3	9.91		17.4	1.91	-	6 +	17.4	16.2	17.1	91	16.4	2;	6.5		1 9	13.4	16.4	14.8	16.3	16.3	14.8	18.1	18.1	12
CaO	12.67		$\perp$	0.5	2.2			3.3	3.7			3.1		_	1.5.	_		2.6		=	7.4	7.7					£. 6	6.9		9.6	1.9	1.1	1.3	7	1.7	1.7	2.7	2.2
12600	_		1		_	_		2.3	_		1.7					2.7			2.6	1.2			-7			1.7	0.7	_				2.9	_		_	_		
Shrinkage 1000C 1200C 1260C		_		- 0			6.1	8	12		- -	_	8.		<del>-</del>	_	_	1.2	_	0.7	_	<del>-</del>	<u>w</u>	LJ.	_	<b>S</b>					1.3			_	9.0	6.0		=
	E	0.7								9	<u></u>	<del>-</del>			<u></u>		0.7			0.3	0.5	1.3	9	9.0	. 9.	9.0		<u>-</u>	, <del>-</del>				0.3		0.7		9.6	0.7
2008.				0.7		· -			0.8	9.0	0.3	0.4	9.0	0.7	0.3		=			<u>.</u>	<u> </u>	_	_	<u>-</u>	6	-		<b>-</b>	<b>⇒</b> ∈		_		_		_		_	
Comp.	A2-15	A2-16	AZ-28	/0t-79	02	BZ 13	BZ-38	8528	BZ-130	BZ-408	BZ-414	BZ-50	1928	BZ-401	BZ-435	BZA-5	82-409	167-791	BZA-4	BZ-46	BZ-403	BZ-433	NZ-400	BZ-415	BZ-410	BZ-419	BZ-36	97.79	20	R7.53	BZ_420	BZ-44	BZ-7	BZ-426	8E-78	BZ-35	BZ-402	BZ-425

Table 2 ... (sheet 6)

<u></u>	٦										<b>-</b> ·	<b>-</b> · ·						_		-	. · <del>-</del>								-		T			-					
Comments																	÷														=								
ide stonite	Ratio	27.50	10.73	18 RG	18 86	16.60	15 80	12.77	67.9	8.03	8.38	7 88	7.17	7.36	÷1.1 9	5.85	5.12	5.22	5 18	CH +	3	1.57	3.45	2 80	277	2.73	2 5.1	2.32	977	S: :	1	* 5			-	I :	=	2.1	7.7
SiO2	-	27.24	2101	25.80	20 63	2.3 88	26.21	2x 15	28.29	25 85	· 72 72	23 0.1	20.43	27 48	2.1.58	22 14	31.33	24.58	25.94	27 17	22.97.	70 P	28.08				27 19	2183	/ × ·	ا ا	7	2.8						7 2	
ZO)S		67.59	5.53	63 07	63.45	63.11	63.27	64.90	02 99	63.89	94.49	62.53	65.83	64.45	E 17	C1 17	67.80	61.86	6.1.30	5.33	62.24	\$5.50	20 99	92 19	51.5	6.3 96	6. 26	3 3 3 3	- ( <u>.</u>	67.01	2	62.40	91.70	3 3		3 3	18 X 3	3 3	62.5
Tion S	Ì			_		0.07					-					0.07						0.07		0.07			0 07												
mol"• Al2O3 Ti		77 8	0 17	5,0	0 33	0.17	0.11	E.E	3.0	27 2	1.0	0.51	0.24	110	11 0	110	0.18	0.23	0 12		:: 10	0.11	o LS	0.17	5 5	- X c		0 32	/70	÷ :	5		;		<u>.</u>	,	57 0	97.0	33.5
7		23.	2.17	0.28	87.0	2.42	0.32	1.65	\$ 12	193	1.64	2 03	5.23	=	1.70	0.27	4.28	5.14	2.65	2 70	1.51		911	0.33	707	0 25	20	T 2	\$ 1	86.0		;		<u> </u>				76 0	1 5
Composition MgO ZA12		7	60	127	61 71	14.08	17.22	11.27	167	13 97	14.07	11 12	7.69	14.26	11 12	17 10	.7.75	7.62	11 78	12.34	11 16:	16 02	7.58	61.11	7.12	13 46	13 16	13.45	77.01	10 20		87 - 78	2 6	- - - - - - - - - - - - - - - - - - -		9:	5 55	77.0	01.0
	L	96 87			_	20.11	19.08	19.07	96 61	20 05	19.71		21.00				19.99	22.14	21.16	22.25	21 92	20.51	22.11	25 28	2117	22 04	22.68	21 10	200	21 97	200	Zv 11	77.77	10.00	5	3 1 1 1 1	25 16	20 07	33.14
ers CaO	_	7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7		0.3		0.4	0.7	0.3	1 90	0 1	10.1		9.6	2	_		0.6	90			6.9			~	90		2				s		_	-	_				
2 Others		66.8	61.9	109	17.19	8.19	8.99	1.79	67.9	1 99	6.99	617	63.8	66.7	65.3	65.6	66.5	63.4	65.1	63.5	61.2	67.1	65	6.1.8	63.2	11911	67.1	64.09	18 07	08 20	2 2	65.77	67.0	2 3	90.00	62 61	69 42	67.38	62 33
Si( 12	ľ			_	·	0.1	_	_	_	_	_	_	_	_		0.7	_		_	_	_	0.1		<u> </u>	_			ا خ -	_	<u>-</u>	1	-	s \	-	c \	د . 		• ·	
AI203 1402		2, 6	70		9.1		0.7	0.7	60	0.2	0.2	6.0	7.	0.7			(1.3	0.7	-		£ 13				63			1.36	<u> </u>	8 .	2		;	80 :	<u> </u>	:	<u> </u>	0.45	0.55
11,0 VI2O				9					_								19	_		9	= (1)	_								_	7	_		_	=				0.016.0
ition ZrO2		- :		19.6					_				_				æ			٠,		_				_		E :		2	1			=======================================	<u> </u>	_			
Composition MgO Ar		0/0	. 6	12.2	12.2	9.7	12.2	9.6	~	9.7	8.6	9.9		9.9		-	5.1			**		11		_					(n./	∵.	١	8.12	_			5.41		3.65	
		2 57	19.2	IR S	18 5	19.3	18.8	Ŧ.	2	101	1.61	100	2	19.1	201	213	18.3	١			21.1	203	20.3	22.8	7.7	21 28	22.1	23.26		~ 	-	25.69	11.77	7	07 17	7.67	23.7		30.62
2600		0 1	6.1			3.1	1.1	2.4	3.6	*	3.4	•	3.2	7.6	10.4	2	2.7	5.7	9	33.7	=	~	+	12.2	5		7.7				1								
2002	l			2 8	26	~	7		6/					t-																									
Shrinkage 1000C 1200C 1260C LCaO		700	<u> </u>	_		<u>.                                    </u>	6.9	.5		-	<u></u>	2.0		6.0	æ /	_	1.4	17	6 2	1.1	7.7	61	0.7	_	7	Ξ	ر د د	- (	7 11	=		5 :	7		=	=	-	5 5	5 C
SOUC IC		3	1.2			0.8 8.0		6.0	70	0.5	0.7	1.5	0.7	0.0	9	0.6	0.2	0.6	0.7	7.1	1.5	_	5.0	1.1	90	<b>%</b>	9.0		-	0.7					÷	0.5			0.0
Comp	-	87.418	BZ-16	BZA-2	BZA-1	BZ-160		BZ-424	BZ-29	87.417	BZ-423	17t-78	11.71	112-42	BZ-122	B13	112,31	BZ-30	87-132	BZ-1340	87.37	814	82-53	BIS	BZ-31	B3-22	B21	B3-23	33-28	B3-27	37.13	<u> </u>	× .	13.6-1.4	121	723	83-18	83.19	83-13

Table 2 ... (sheet 7)

culs	_							_			-	
Comments			L			_						_
Diopside	Wollastonite	Ratio		=:								
	Exerss		35 17	30 19	28 49	46.38	36.34	30 07	31 05	27 05	23 15	28 8U
	SiCi2		167.73	63.22	64.27	73.37	68.42	65.20	65.52	09.60	61.60	64.52
	Tic 2											
o "Jou	VI21)3		l	0.27								
_	_			0.25								
Compositi	MgO Zat		2 79	2 63	2.92	76.0	17.0	68.0	1.42	0.89	0.88	0.55
	- (학)		28.79	31.64	32.59	25 13	30.13	33.27	32.77	35.03	37.18	7.1
	Others				0.3			0.5	7	0.3	0.5	
	Si(1)		67.25	98 59	65.5	73.28	67.59	65.3	66.4	64.1	62.3	£.13
	TiO2											
wi"o		1		0.47								
				0.51							0.1	
Composition	C <sup>H</sup> W			1.78								
J	ÇiiÇ		26 68	29.82		23.43	27.76	31.1	=	33	35.1	31.93
	1000C 1200C 1260C 1 CaO				3.2				1.8	1.3	7.7	_
	200C							1.1		f		
Shrinkage	GIOC 1		0.3	£.			1.7	7.		8.1	1.7	9.11
	SOUC 1							0.1		9.0	0.5	
	Comp		193-17	B3-15	E32	<b>B3-3</b> 2	B3-31	E25	E31	E24	E23	B3-30

Table 3 (sheet 1)

INEO	otal Excess	-37.16		39 -15 67	6.5 -2.74		23 -1.43		21 -1.28			_			166 0.00				136 0.26	235 0 39	× ×	172 0 11	198 0.54								30 0 72 226 1 72 27 1.24								
	SiO2 lotal	-	13	٥	o;	5.8	~	91	=	6	17	36		7.1	112	18	163	25	98	5,		113	137	18	162	133		: 3	9/	64/ 64/	91	193	92 18	16 16 18 193	193 193 132 132 133 133	193 193 132 132 162 163	193 193 132 132 162 163	193 193 193 193 195 195 195 195 195	19.2 16.2 16.2 16.2 16.2 16.2 16.2 16.2 16
Solubility	Oğw				2	,	~	~	~	•	-	~	•~	~	<b>-</b> 7	**.	-,	'n		ır.	×			٠٠.					u .v										
	ر ديون 			30				2		9	_			20			<del>-</del>		_		×				79								·						
	Sit 12	63 63	19 SD	25 51	60 6.8	99 19	62 93	65.75	67.34	67.22	62.57	39 16	54 22	64.18	68 12	£09	64 52	57.07	65.20	59 63	73.37 X	63 60	91.60	64 112	5937	57.35	03.0	62.10	54.03	54.03	62.10 54.03 67.40 65.52	65.76 65.76 65.76 65.76	65.40	62.10 54.03 67.40 65.52 69.17 69.17	62.10 54.03 67.40 65.52 65.76 66.70	62.70 65.71 65.72 65.73 65.74 65.75 65.73	62.10 67.10 65.52 65.76 69.17 69.17 65.22 68.88	62.10 54.13 65.14 65.14 65.14 65.14 65.14 65.13 65.13 65.13 65.13	62.10 54.13 65.14 65.14 65.14 65.14 65.14 65.14 65.14 65.14 65.14
	Alzoa Troz	32.28	25.12	15 67	3.55	2.48	0.13	0.19	0.13	0.19	0.19	61.0	0.31	0.19	0.24	61 10	018	0.25	0.29	0.54	81.0	0.29	0.29	0.25	0.35	6.35	71.0	2 0	8/0	0 /2	0.12 0.12 9.23	0 / 9 0.12 0.13	6.12 6.13 0.12	0 18 0 0 18 0 0 18 0 19 0 19	0 / 8 0 12 0 12 0 13 0 15 0 15	0.18 0.12 0.12 0.39 0.42 0.42	0 13 0 12 0 12 0 13 0 15 0 14 0 14 0 18	0 13 0 12 0 13 0 13 0 13 0 13 0 18	0 18 0 12 0 13 0 13 0 13 0 13 0 18
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	C <sub>ii</sub> C	2.9	: /9.7	20.5	1016		17.6	11.9	13.4	15.1	18.9	22.3	263	17.4	27.76	20.7	31.93	23.5	31.1	×	23.43	į.	35.1	19.3	37.1	10.1	0.00	42.79	* * * * * * * * * * * * * * * * * * * *	<u>'</u>	3.5	8. 3. 3. 3. 1.	31 31 2 2 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	16.5 31 18.1 . 24.99	18.1 24.99 18.1 18.1 26.68	31 18.1 24.99 18.1 26.68 29.82	18.1 18.1 24.99 18.1 26.68 29.82 29.82 16.3	18.1 18.1 18.1 18.1 26.68 29.82 29.82 16.3	16.7 18.1 18.1 18.1 18.1 26.68 29.82 16.3 16.3
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Table 3 ... (sheet 2)

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M <sub>E</sub> O Zr	01.5	7.76	2.92	3.42	3.67	3.60	7.42	7.78	7.58	6.83	7.43	£6.9	7.43	5.58	7.17	5.8.1	5 42	7.80	5.55	7.26	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	14.17	1.00	15.19	7.15	9.68	14.05	13.92	7.01	15.36	11.12	14.21	15.52	6.71	9/ /	7 30	07.7	;
CaO MgO ZrO	33 14	35.00	32.59	35.10	36.35	33.96	21.17	19.99	11 65	26.57	20.02	27.07	33 95	34.36	25.58	13 92	26.61	28.69	25.16	25 25	\ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	12.76	11 11	9.12	27.18	60.81	12.98	13.01	36.13	11.71	13.05	13.37	13.83	37.04	27.50	13.74	17.114	,
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Table 3 .. (sheet 3)

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Table 3 ... (sheet 4)

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		0.07	9.07	6.00	0.00
3.12 3.06 2.44 5.34	23.23 2.23 2.24 2.24 2.24 2.24 2.24 2.24	2.13 2.13 2.14 2.14 2.14 2.14 2.14 2.14 2.14 2.14	2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.2.	2.23 2.23 2.23 2.23 2.23 2.23 2.23 2.23	2.12 2.13 2.14 2.14 2.15 2.15 2.15 2.15 2.15 2.15 2.15 2.15
2.0 2.0 3.0 3.0 3.0 3.0 3.0 3.0 3.0 3.0 3.0 3	S	8 7 7 8 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9	8 4 4 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8		67.4 0.5 62.6 0.3 61.4 0.5 61.9 0.5 64.9 0.4 64.7 0.3 64.7 0.3 66.1 0.4 66.1 0.4 66.1 0.4 67.7 0.4 66.1 0.4 66.1 0.4 67.7 0.4 66.1 0.4
0.1	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	000000	0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0		
	9.6 5.1 9.7 5.4 9.7 5.4	- 52 55 52 54 5 5 5 5 5 5 5 5 5 5 5 5 5 5	- U444 - U - U - U - U - U		•
	200	7.6.6.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.8.			

Table 3 ... (sheet 5)

Comp.	<u> </u>	Ç <sub>i</sub> M	Z()17	VIZO3	TiO2	SiO2	Others	<u>ာ</u>	Çg W	2r()2	Al2O3	1102	Sit)2	C <sub>2</sub> C	MrO	SiO2	Total	Excess
BZ-4150B	15.3	11.5	1.1		0.7	25	6.0	16.08	L	L	L	0.0	62 00					12.76
B3-22	21.28	9.34	0.54	_		66.17		22.04	13.46	0.25			63.96	62	57	174	29.3	12.90
B3-25	28.15	9.22	•	_		59.53		29.08	_				57.40	8	33	133	214	12.98
B3-23	23.26	9.33	0.3	_		64.09		24.10	_	= 0			66 19	7	31	101	176	12 99
BZ-38	17.2	11.2	6.3	0.7	_	63.5	7.0	18.06	16.36		0.12		61.25	9	32	09	132	13.05
B3-26	30.5	11.6		_	· ·	56.95		31.39	_		0.35		54.74	19	=	149	254	13.17
BZ-4150.1	15.3	11.5				64.5			_	_		0.07	63.10					13.25
BZ-4150	15.2	11.7			5	65.3	0.5	_	16.97	3.51	0.00	0.07	63.54	17.00	14.00	37.00	<b>K9</b>	13.40
A2-16	12.4	60 01	2.23	-				12.90	_		_			÷		113	_	•
110	7.7	11.1	53		2.7			14.97	_		0.00	197	64.14	22	28	2	95	
12-435	9.91	11.3	5.3	9.0		6.19	0.7	17.36	_	_	0.35		63.34	23		89		
B.3-2-1	25.53	9.73		0.58		61.62		26.35	13.97		0.33		59 36	37		185		
ra Ea		11.3	5.7	170	_	67.3	03	14.24			_	120	65 79			St	73	
BZ-1150C		11.9	7.5	7	10	63.0	8.0	16.27	17.33	3.58		0.07	62.63					13.74
37.433	16.2	11.5	vi	0.2		65.8	0.9	16.84		_	0.11		63.85					13.96
07	15.4	11.6	æ;	0.2	_	68	0.3	16.16	_	_	9.11	0.73						17.03
818	22.8	10.2	03	0.3	=	64.8	0 2	23.28	_		0.17	0.07				137	232	071
Sa	11.3	117	5.4	0		99	10	14.82	_	233	0.00	1.46	64.23	7.7				14.27
DIO	15.7	11.7	6+	7: 0:	2.7	64.7	0.3	16.26	_	_	0.00	1.96						
BZ-1-6	17.2	11.9	3.4	-			0 +	17.71	_		_		62.86			50	3	
BZ-437	15	17.7				65.7		15.60	_		_		63.79					11.71
BZ-410	15.9	12.1				_		16.47	17.44				63.53					
2	16.1	12.2	,		8/			17.11	_	2.56	_	1.32		21	20		16	
BZA-5	17.1				_		=	17.63	_		5 0.57	0.07	63 21	8		4	æ	15.04
BZA-4		11.9	3.1	0.7		658			_		_		63.47	71			<u>&amp;</u>	_
7	16.3	125			_	633		_			_	0.73	61.50	72			103	
BZ-410.1	16.1	126	3		7	979	6.7		_	_	90.0	0.07	62.31					155
B2-110C	16.2	126	3		70		6.8	16.82	_		0.00	0 0 7	62.33					1364
814	20.3	7:17									0.1	0.07				_		
2	15.9	7	1.87				0.7	_	_	7 0.87		0.65	65.10					
A2-14	25.3	11.66				60.74	_	25.70	_	_			57.59		_			_
BZA-2	18.5	12.2	9.0			1.99		_	_				63 07					16.43
877-3	18.6	12.4			_	65.8	0.3	_		_			62.47					
3ZA-1	18.5			9.0		67.1		_	_				63.45	25	25	ઙ	=	
A2-7	23.37	_		_		61.98		23.79	_	_			58.89			_		
B13	21.3	12.3			0.7		5 0.3		8 17.10			0.07						
B4	18.8		0.1			8.99			_	_			63.27					
A2-15	12.67	٠				72.25		12.94	_				68.89					
A2-9	21.44	12.96		1.49	_	43.66		31.5	_		0 0		40.00		_	_		
					_	3	_	41.30	_	_	70.0		7		_	_	378	_

Table 3 .... (sheet 6)

Comp.	CaC	NgC.	. ZCD2	AIZO3	Ti()2	SiO2	() there	CaO		MgO ZrO	_	A1203	Ti02	SiO2	<del>-</del>	C <sub>E</sub> O	Οĝ	SiO2	Total	Excess
	0 31	1	٦	L		-		L	1111	18 50	0.83	0 22	0 71	11 62 54	12	=	30	200	071	17.5
2 5	1 0		- ×	-				0.2	19.1	18.24	0.36				12	Ŧ	45	110		_
750	17.15	12.6				68.33		_	17.67	17.75				64 58	80	38	9	117	195	_
	w/.	13.5		0.7		65		0.7	18.57	18.85	0.78	<u>-</u>	0.70	60.99	66	34	36	63	_	17.96
2.18	26.29	13.01		_		56.9	90		26.85	18.48		0.37		34.30	30	17	37	161		
173	22	13.2	0.1		0.1	1 63.5		0.2	22.03	18.39	0.03	0.11	0.07		33	\$	£.	20	161	
2.11	24 28	13.24	0.08			Ş	32		21.18	18.57	0.0	9.1		56 77	11	72	9[	119	_	_
	19.78	14.54	0.66	5 2.57		9	32		66.61	20.44	0.30	1.43	_	57.84	**	36	37	77		_
BZ4	18.3	13.7	0.3		2 0.1	1 66.8	85.	9.0	18.29	19.03		0.11	<u> </u>		33					18.81
22	18.3	13.8			2 0.1		5.1	1.3	13.15	19.36	_	_		0.07 61 64	2					_
2.5	18.71	13.78	_	_	- <u>-</u>	65.1	69		18.85	19.29		_	_	69.19	69	¥	5	150	258	_
27.	18	13.8			=	<b>%</b>		0.7	18.09	19.29		0.00	~	62.47	11	-			_	
2	19.7		0		~	- 3	-	0.4	19.05	19.60	0.03	0.11	_	61.20	20	9	S	#		_
3.6	16.86	14.24		_	7		33		17.15	20.15	0.54	0.12	<u></u>	62 03	03	¥	ಹ	181		_
27.7	11 58		1.58	_		20	43		11.78	20.55	0.73	0.08		99	9H 99	35	72	6	_	
Biti					2 0.1		3.6	0.2	15.63	20.65	0.18	<u> </u>		0.07 63.	63.36	37	ŧ	2	_	_
	00/		0.0				1.2	10	19.71	20.80	0.03		_	59	59 34	<del></del>	<b>**</b>	ž	_	_
	316		_			20	85	_	21.58	21.73	0.03	_	~		55.80	7	<b>:</b>	92	2 176	_
757	20 02			0.2	2	9	2.6		20.79	21.04		0.1	_	\$8	98 196	62	2	_		_
117	14.2		0.0				69.1	0.7	14.14	21.47	9.08	_	_	0.07 64	64.22	32	45			
1	8.91				2 0.1		67.1	0.2	16.49	21.71	0.13	_			61.48	30	<del></del>	•		_
12-10	16.22		_	0.49		99	11		16.18	21.93		0.27	_	-	61.62	7	25	122		
12-13	14.87	16.01			_	- 66	19.99		14.89	22.31	_	_	9	2	61.31	Ç	2			
A2-22	9.36		_	3 0.33	13	7	8°7.		9.42	22.88	_	_	<b>∞</b>	-6	67.14	36	75		`	_
1 72	26.62				_	56	56.58		25.66	22.87	_				20.02	32	8			_
6/6	13.9					0.1	68.7	10	13.65	22.93		0.03		0.07 62	62.97	\$	Ó¥			_
A 2-20	11.58				7.	28.	68.19		11.7	23.30	_	_	7	હ —	64.35	<u></u>	9		)Z   [29]	262 22 66
07.3	24.45	16.81			0.5	56	56.18		24.31	23.25	0.02			<u>~</u>		77	17	•		_
A 2.78	2.07			_	15	78	78.07	_	2.08	23.93		_		-	73.10 X		×	×	×	_
2 2	8				.7	<b>~</b>	61.7	0.2	17.72	23.29	_	_	_	₹ -	58.84	32	7			
021	23.02		_		0.74	28	56.82		23.56	23.79	_	2 0.10	9	-	\$2.23	35	73	_	112 2	
	18.8				12	_	63	0.2	18.30	21.23	1 0.13		=	<u>د.</u>	57.23	30	35			159 24.00
970	-					0.1	99	0.5	14.78	24.65		3 0.03		0.07 60	60.31	35	19			
A2.13	16.55		_	_			63.56	_	16.37	24.76	0.02	2 0.18	80	<u> </u>	28.67	47	99	<u> </u>		
A2.30	15.75	18.71			15.0	- 63	63.68		15.89		_	0.22	77	<u>~</u>	58.82	33	3	_		_
42.23	18 50		0.05	_	18	_	60.2	_	18.37	25.82	_	_	9.	.5	55.53	35	÷		2 2	
A2.26	8.12	19.26			0 29	39	68.65	-	8.15	26.90	_	_	91	<u>څ</u>	6434	31	97	_		311 26.29
A2-32	6.36		_	0.36 0.	23	7.	73.09		6.23		0 0.16		12	٠	8.79 X		×_	×	×	Ze-11
A2-27	677	_			2.1		71.14	_	6.70			15 0.13	<u> </u>	•	69.89	7-1	67		=	192 Z6 44
				_	0.3	-	41.40	_	32.11	36.47	•	_	=	_	- 11	ž				

Table 3 .... (sheet 7)

		Composition	lion	w196				Conipos	ilion	mole,		-		Calculation			
Comp.	CaO MgO	Og W	2012	AIZU3 TRO2	SiO2	Others	Ono	Og M	202	A1203	1702	SiO2	ت د	MgC	SiO2	Fotal	Excess
01-21	St 81	1		0.54	58.71		18 29			0,0		77.73	1	- 1		1	1
A2-21	13.74		_		- F		13.51			_		28 00					
A2-31	8.45		Ī		67.62		8 26					2 2					
A2.29	1.16				71.24		7.7					2 2					
A2-33	19.1				20.01		5					70.00		_			
12-21	13.62				26.19		14.25					09.00					
12-25	10.99		_		62.36		79.07			_		25.70					
A2-35	8.88		_		61.12		8.56					\$7.71					
41-34	6.63	26.2	0.8	0.23	64.85		6.37	35.01	0.35	0.12		58.75	= =	0//	2 %	6 2	33.01
765	3.9				57.78		3.62					50.03					
		i	:		J		11111			1				•			_

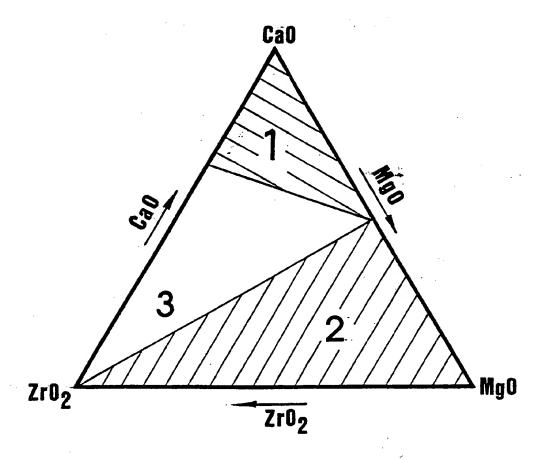
### **CLAIMS**

- 1. A refractory fibre for which a vacuum cast preform of the fibre has a shrinkage of 3.5% or less when exposed to 1260°C for 24 hours and comprising CaO, SiO<sub>2</sub>, MgO, optionally ZrO<sub>2</sub>, optionally less than 0.75mol% Al<sub>2</sub>O<sub>3</sub>, any incidental impurities amounting to less than 2mol% in total, and in which the SiO<sub>2</sub> excess (defined as the amount of SiO<sub>2</sub> calculated as remaining after the above named constituents are crystallised as silicates) exceeds 21.8mol%, with the proviso that, if the amount of CaO is greater than the sum of the amount of MgO and twice the amount of ZrO<sub>2</sub> the calculated ratio of diopside to wollastonite does not lie in the range 1.8 to 5.25.
- 2. A refractory fibre as claimed in claim 1 in which the incidental impurities include TiO<sub>2</sub> in an amount less than 1.25mol%, preferably less than 0.8mol%.
- 3. A refractory fibre as claimed in claim 1 in which the incidental impurities include Na<sub>2</sub>O in an amount less than 1.0wt%, preferably less than 0.5wt%, more preferably less than 0.3wt%.
- 4. A refractory fibre as claimed in claim 1 in which the incidental impurities include Fe<sub>2</sub>O<sub>3</sub> in an amount less than 1.0wt%, preferably less than 0.6wt%.
- 5. A refractory fibre as claimed in claim 1 in which Al<sub>2</sub>O<sub>3</sub> is present in an amount less than 0.5mol%
- 6. A refractory fibre as claimed in any of claims 1 to 5 and having a composition in which the amount of CaO is less than the sum of the amount of MgO and twice the amount of ZrO<sub>2</sub>.
- 7. A refractory fibre as claimed in claim 6 and in which the amount of MgO is greater than the amount of CaO.
- 8. A refractory fibre as claimed in claim 7 characterised in that a vacuum cast preform of the fibre has a shrinkage of less than 3.5% when exposed to 1300°C for 24 hours.
- 9. A refractory fibre as claimed in any of claims 1 to 8 and which is saline soluble.

- A saline soluble refractory fibre as claimed in claim 9 in which the excess MgO (defined as the amount of MgO less the sum of the amounts of  $ZrO_2$  plus  $Al_2O_3$ ) exceeds 10mol%.
- 11. A saline soluble refractory fibre as claimed in claim 10 in which the excess MgO exceeds 11.3mol%
- 12. A saline soluble refractory fibre as claimed in claim 11 in which the excess MgO exceeds 15.25mol%
- 13. A method of providing a saline soluble refractory fibre for use at elevated temperatures comprising selecting a fibre as claimed in any of claims 1-12.
- 14. A saline soluble fibre characterised in that a vacuum cast preform of the fibre has a shrinkage of 3.5% or less when exposed to 1260°C for 24 hours.

1/1

Fig.1



## INTERNATIONAL SEARCH REPORT

PCT/GB 94/00053

A. CLASSIFICATION OF SUBJECT MATTER IPC 5 C03C13/00				
According to international Patent Classification (IPC) or to both national classification and IPC				
B. FIELDS SEARCHED  Minimum documentation searched (classification system followed by classification symbols)	, <del></del>			
IPC 5 CO3C				
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched	ed .			
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)	·			
DO THE CONTROL TO BE BUILDYANT				
C. DOCUMENTS CONSIDERED TO BE RELEVANT  Category ' Ottation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.			
Category' Citation of document, with indication, where appropriate, of the relevant passages				
P,L, WO,A,93 15028 (THE MORGAN CRUCIBLE COMPANY PLC) 5 August 1993 see the whole document	1-14			
X WO,A,87 05007 (MANVILLE CORPORATION) 27 August 1987 cited in the application see claims 1-5	1-14			
WO,A,89 12032 (MANVILLE SALES CORPORATION) 14 December 1989 cited in the application see claim 1	1-14			
X WO,A,92 09536 (PAROC OY AB) 11 June 1992 see claim 1	1-5,9			
Further documents are listed in the continuation of box C. X Patent family members are listed in ann	nex.			
*Special categories of cited documents:  "T" later document published after the internation or priority date and not in conflict with the considered to be of particular relevance  "A" document defining the general state of the art which is not considered to be of particular relevance  "I later document published after the internation or priority date and not in conflict with the cited to understand the principle or theory to invention."	: application but			
earlier document but published on or after the international filing date  "X" document of particular relevance; the claimed invention carnot be considered novel or cannot be considered to				
which is cited to establish the publication date of another citation or other special reason (as specified)  "Y" document of particular relevance; the claim cannot be considered to involve an inventive	ned invention we step when the			
other means ments, such combination being obvious to a document rephished prior to the international filing date but in the art.	a person skilled			
later than the priority date claimed  *&' document member of the same patent famil  Date of the actual completion of the international search  Date of mailing of the international search				
2 1. 04. 94				
Name and mailing address of the ISA  Authorized officer  Authorized officer				
European Patent Office, P.B. 5818 Patentiaan 2  NL - 2280 HV Rijswijk  Tel. (+31-70) 340-2040, Tx. 31 651 epo nl.  Fax: (+31-70) 340-3016  Reedijk, A				

#### INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 94/00053

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